EXHIBIT 7

INTERNATIONAL

Designation: E 679 – 91 (Reapproved 1997)

An American National Standard

Standard Practice for Determination of Odor and Taste Thresholds By a Forced-Choice Ascending Concentration Series Method of Limits[1]

This standard is issued under the fixed designation E 679; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

The obtaining of odor and taste thresholds requires the sensory responses of a selected group of individuals called panelists. These thresholds may be determined in order to note the effect of various added substances on the odor and taste of a medium. They may also be determined in order to characterize and compare the odor or taste sensitivity of individuals or groups.

It is recognized that precise threshold values for a given substance do not exist in the same sense that values of vapor pressure exist. The ability to detect a substance by odor or taste is influenced by physiological factors and criteria used in producing a response by the panelist. The parameters of sample presentation introduce further variations. Thus, the flowrate of a gaseous, odorous sample has an influence on the detectability of an odor. However, a concentration range exists below which the odor or taste of a substance will not be detectable under any practical circumstances, and above which individuals with a normal sense of smell or taste would readily detect the presence of the substance.

The threshold determined by this practice is not the conventional group threshold (the stimulus level detectable with a probabililty of 0.5 by 50 % of the population) as obtained by Practice E 1432, but rather a best estimate not far therefrom. The bias of the estimate depends on the concentration scale steps chosen and on the degree to which each panelist's threshold is centered within the range of concentrations he or she receives. The user also needs to keep in mind the very large degree of random error associated with estimating the probability of detection from only 50 to 100 3-AFC presentations.

1. Scope

1.1 This practice describes a rapid test for determining sensory thresholds of any substance in any medium.

1.2 It prescribes an overall design of sample preparation and a procedure for calculating the results.

1.3 The threshold may be characterized as being either (*a*) only *detection* (awareness) that a very small amount of added substance is present but not necessarily recognizable, or (*b*) *recognition* of the nature of the added substance.

1.4 The medium may be a gas, such as air, a liquid, such as water or some beverage, or a solid form of matter. The medium may be odorless or tasteless, or may exhibit a characteristic odor or taste per se.

1.5 This practice describes the use of a multiple forced-choice sample presentation method in an ascending concentration series, similar to the method of limits.

1.6 Physical methods of sample presentation for threshold determination are not a part of this practice, and will depend on the physical state, size, shape, availability, and other properties of the samples.

1.7 It is recognized that the degree of training received by a panel with a particular substance may have a profound influence on the threshold obtained with that substance (**1**).[2]

1.8 Thresholds determined by using one physical method of presentation are not necessarily equivalent to values obtained by another method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1292 Test Method for Odor in Water[3]

E 544 Practice for Referencing Suprathreshold Odor Intensity[4]

E 1432 Practice for Defining and Calculating Individual and

[1] This practice is under the jurisdiction of ASTM Committee E-18 on Sensory Evaluation of Materials and Products and is the direct responsibility of Subcommittee E18.04 on Fundamentals of Sensory.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this practice.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 15.07.

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Group Sensory Thresholds from Forced-Choice Data Sets of Intermediate Size[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *sample*—a material in any form that may or may not exhibit an odor or taste, depending on the amount of odorous or sapid components that it may contain.

3.1.2 *medium*—any material used to dissolve, disperse, or sorb odorous or sapid material whose threshold is to be measured.

3.1.3 *blank sample*—a quantity of the medium containing no added odorous or sapid material.

3.1.4 *test sample*—the medium to which an odorous or sapid material has been added at a known concentration.

3.1.5 *detection threshold*—the lowest concentration of a substance in a medium relating to the lowest physical intensity at which a stimulus is *detected* as determined by the best-estimate criterion.

3.1.6 *recognition threshold*—the lowest concentration of a substance in a medium relating to the lowest physical intensity at which a stimulus is *recognized* as determined by the best-estimate criterion.

3.1.7 *best-estimate criterion*—an interpolated concentration value, but not necessarily the concentration value that was actually presented. In this practice it is the geometric mean of the last missed concentration and the next (adjacent) higher concentration.

3.1.8 *panelists*—individuals whose odor or taste thresholds are being evaluated, or who are utilized to determine the odor or taste threshold of the substance of interest.

3.1.9 *ascending scale of concentrations*—a series of increasing concentrations of an odorous or sapid substance in a chosen medium.

3.1.10 *scale steps*—discrete concentration levels of a substance in a medium, with concentrations increased by the same factor per step throughout the scale.

3.1.11 *3-alternative forced choice (3-AFC) presentation*—a set consisting of one test sample and two blank samples (as applied to this practice).

3.1.12 *geometric mean*—the *n*th root of the product of terms. In this method, the terms are concentration values.

4. Summary of Practice

4.1 A series of test samples is prepared by dispersing the substance whose threshold is to be determined in the medium of interest. This concentration scale should increase in geometric increments so that any two adjacent concentration steps are separated by a constant factor. At each concentration step, two blank samples consisting of the medium only are made available to the panelist. The blank and test samples are encoded so that there is no visual, audible, tactile, or thermal difference between the samples other than code designators (**2**).

4.2 The panelist starts at the lowest concentration step, which should be two or three concentration steps below the estimated threshold. Each sample within the set of three is compared with the other two.

4.3 The panelist indicates which of the three samples is different from the other two. A choice must be made, even if no difference is noted, so that all data can be utilized.

4.4 Individual best-estimate values of threshold are derived from the pattern of correct/incorrect responses produced separately by each panelist. Group thresholds are derived by geometrical averaging of the individual best-estimate thresholds.

5. Significance and Use

5.1 Sensory thresholds are used to determine the potential of substances at low concentrations to impart odor, taste, skinfeel, etc. to some form of matter.

5.2 Thresholds are used, for example, in setting limits for air pollution, in noise abatement, in water treatment, and in food science and technology.

5.3 Thresholds are used to characterize and compare the sensitivity of individual or groups to given stimuli, for example, in medicine, in ethnic studies, and in the study of animal species.

6. Preparation of Concentration Scale

6.1 The concentration levels of the test substance in a medium should begin well below the level at which the most sensitive panelist is able to detect or recognize the added substance, and end at (or above) the concentration at which all panelists give a correct response.

6.2 The increase in concentration of the test substance per scale step should be by a constant factor. It is desirable to obtain a scale step factor that will allow the correct responses of a group of nine panelists to distribute over three to four concentration steps (see Appendix X1). This will allow more accuracy in determining the threshold value based on the geometric mean of the individual panelists.

6.3 Good judgment is required by the person in charge in order to determine the appropriate scale step range for a particular substance. This might involve the preparation of an approximate threshold concentration of the odorous or sapid substance in the medium of choice. The concentration of the substance may be increased two to three times for odorants or 1.5 to 2.5 times for sapid substances depending on how the perceived intensity of odor or taste varies with the concentration of the substance providing the sensory response. Thus, if x represents an approximate odor threshold concentration, then a series of concentration steps would appear as follows if a step factor of "3" were used:

$$\ldots x/27, x/9, x/3, x, 3x, 9x, 27x \ldots$$

6.4 In actual practice, the various concentrations are obtained by starting at the highest concentration and diluting three times per step, thus providing a series of dilution factors, "V_i" being the initial volume:

$$\ldots 729V_i, 243V_i, 81V_i, 27V_i, 9V_i, 3V_i, V_i \ldots$$

6.5 At each selected concentration or dilution, a 3-AFC sample set consisting of one test and two blank samples is presented to panelists in indistinguishable fashion (**3**). It is desirable to have all samples prepared and ready for judging before the evaluation session begins. (Reference (**2**) contains

sound practices for coding the samples, rotating the positions of these test and blank samples as the test proceeds, etc.)

6.6 If the samples are arranged in a left-center-right, or an above-center-below order, care must be taken that the test sample is presented in one third of the presentations in the left (top) position, one third in the center position, and one third in the right (bottom) position to eliminate positional bias.

6.7 If only one sample at a time is available, the test and blank samples may be presented one after another in units of three presentations, with the test sample being randomized to be the first, the second, and the third, and requesting the response after all three samples in the set have been presented. Better results, however, are obtained if the test and the two blank samples are available for a direct comparison, so that the panelist may sniff or taste back and forth at ease until a decision is reached.

7. Judgment Procedure

7.1 The panelist begins judging with that set which contains the test sample with the lowest concentration (highest dilution) of the odorous or sapid substance, takes the time needed to make a selection, and proceeds systematically toward the higher concentrations.

7.2 Within each set, the panelist indicates that sample which is different from the two others (detection threshold) or which exhibits a recognizable odor or taste of the substance (recognition threshold). If the panelist cannot readily discriminate, a guess must be made so that all data may be utilized.

7.3 The judgments are completed when the panelist either (*1*) completes the evaluation of all sets of the scale, or (*2*) reaches a set wherein the test sample is correctly identified, then continues to choose correctly in higher concentration test sample sets.

8. Data Evaluation

8.1 The series of each panelist's judgments may be expressed by writing a sequence containing (0) for an incorrect choice or (+) for a correct choice arranged in the order of judgments of ascending concentrations of the added substance.

8.2 If the concentration range has been correctly selected, all panelists should judge correctly within the range of concentration steps provided. Thus, the representation of the panelists' judgments as in 8.1 should terminate with two or more consecutive plusses (+).

8.3 Because there is a finite probability that a correct answer will occur by chance alone, it is important that a panelist continues to take the test until there is no doubt by that person of the correctness of the choice.

8.4 The best-estimate threshold concentration for the panelist is then the geometric mean of that concentration at which the last miss (0) occurred and the next higher concentration designated by a (+).

8.5 The panel threshold is the geometric mean of the best-estimate thresholds of the individual panelists. If a more accurate threshold value of an individual panelist is desired, it may be obtained by calculating the geometric mean of the best-estimate threshold of all series administered to that person.

9. Report

9.1 Successful completion of the foregoing procedure provides either the detection or recognition threshold of the substance in the medium of interest in accordance with this practice.

9.2 The threshold value is in concentration or dilution units appropriate for the substance tested (**4**).

9.3 For enhanced understanding of the threshold results, the following information is recommended:

Threshold of:
Procedure: ASTM Practice E 679 (Rapid Method)
Presentation:
Number of scale steps:
Dilution factor per step:
Temperature of samples:
Panelist selection:
Number of times test given:
Type of threshold (detection or recognition):
Best-estimate threshold:
  Individual:
  Panel:

9.4 Refer to Appendix X1 for an example of the calculation required and reporting.

10. Precision and Bias

10.1 Because sensory threshold values are functions of sample presentation variables and of individual sensitivities, interlaboratory tests cannot be interpreted statistically in the usual way, and a general statement regarding precision and bias of thresholds obtained by this practice cannot be made. However, certain comparisons made under particular circumstances are of interest and are detailed below.

10.2 When 4 panels of 23 to 35 members evaluated butanol in air (**5**), the ratio of the highest to the lowest panel threshold was 2.7 to 1; when the same panel repeated the determination on 4 days, the ratio was 2.4 to 1. For 10 panels of 9 members evaluating hexylamine in air, the ratio was 2.1 to 1.

10.3 When 26 purified compounds were tested for threshold by addition to similar beers by 20 brewery laboratories (each compound was tested by 2 to 8 laboratories), the ratios of the highest to the lowest panel threshold varied from less than 2.0 to 1, to 7.0 to 1 or more (**6**). The lowest variability was found with simple compounds of high threshold (sugar, salt, ethanol), and the highest with complex compounds of low threshold (eugenol, hop oil, geosmin).

10.4 When 14 laboratories determined the threshold of purified hydrogen sulfide in odorless air (**7**), the ratio of the highest to the lowest laboratory threshold was 20 to 1. Interlaboratory tests with dibutylamine, isoamyl alcohol, methyl acrylate and a spray thinner for automobile paint gave somewhat lower ratios. Although the methods used vary somewhat from this practice, the results are comparable.

10.5 A discussion of the likely bias of results by this practice compared to a true threshold can be found in references (**5**), (**8**) and (**9**).

11. Keywords

11.1 air pollution; ascending method of limits; odor; panel; sensory evaluation; taste; threshold; water pollution

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE

X1.1 The odor threshold of an odorous air sample was to be determined.

X1.2 Six different concentrations of the odorous sample in air were prepared. Each of these was presented in conjunction with two samples of nonodorous air. The concentrations were increased by a factor of three per concentration step. Nine randomly selected panelists participated. Each proceeded from the lower to higher concentrations. At each concentration level, panelists compared the three samples—two blanks and one diluted odorous sample—and indicated which sample was different from the other two.

X1.3 The following results were obtained (see Table X1.1):

X1.4 Details of calculation are as follows:

X1.4.1 For Panelist 1, the best-estimate threshold is $\sqrt{135 \times 45} = 78$, or at a dilution by a factor of 78 (one volume of the odorous air sample diluted with nonodorous air to occupy 78 volumes in total). For Panelist 2, the threshold is at $\sqrt{1215 \times 405} = 701$.

X1.4.2 Panelist 4 missed at the highest concentration, where the dilution is only by a factor of 15. It is assumed that he would have been correct at a higher concentration level, where the dilution would have been a factor 15/3 = 5.

TABLE X1.1 Example of Odor Threshold

NOTE 1—This example has been selected to represent both extremes. Panelist 4 missed even at the highest concentration. Panelist 6 was correct even at the lowest concentration and continued to be correct at all subsequent higher concentrations.

Panelists	Judgments[A] Dilution Factors (concentrations increase →) 3645	1215	405	135	45	15	Best-Estimate Threshold (BET) Value	$\log_{10}$ of Value
1	0	+	+	0	+	+	78	1.89
2	+	0	+	+	+	+	701	2.85
3	0	+	0	0	+	+	78	1.89
4	0	0	0	0	+	0	9	0.94
5	+	0	0	+	+	+	234	2.37
6	+	+	+	+	+	+	6313	3.80
7	0	+	+	0	+	+	78	1.89
8	+	0	0	+	+	+	234	2.37
9	+	0	+	+	+	+	701	2.85
Group BET geometric mean							$\Sigma\log_{10}$ → / 209 ←	20.85 / 2.32
Standard deviation								0.81

[A] "0" indicates that the panelist selected the wrong sample of the set of three. "+" indicates that the panelist selected the correct sample.

X1.4.3 Consequently, an estimate of his threshold is $\sqrt{15 \times 5} = 9$. The underlying assumption is that since the thresholds of the other panelists were within the presented scale range, his threshold should not be far away from the range if he belongs to the same statistical population. If the test were to establish the sensitivity of the panelists, this panelist would have been retested, with a scale range extended to the right of the results in Table X1.1.

X1.4.4 Panelist 6 represents the opposite extreme. The estimate is based on the assumption that a miss would have occurred at a dilution of $3 \times 3645 = 10\ 935$; the best-estimate threshold is then $\sqrt{10\ 935 \times 3645} = 6313$.

X1.4.5 In Table X1.1, dilutions change exactly by a factor of three per scale step. Experimentally, small deviations from such equal spacing occur, and the actual dilutions or concentrations should be used in calculating the best-estimate thresholds from two adjacent values in the table.

X1.5 *Report*—The report shall include the following information:

Odor threshold: Odorous Air Sample XX
Procedure: ASTM Practice E 679
Presentation: at 500 ml/min (dynamic dilution olfactometer)
Number of scale steps: 6
Dilution factor per step: 3
Temperature: 25°C (room and samples)
Panelist selection: random
Number of panelists: 9
Type of threshold: detection
Best-estimate threshold:

$$Z_{OL} = 209$$
$$\log_{10} Z_{OL} = 2.32$$
$$\text{Standard log deviation} = 0.81$$

NOTE X1.1— The symbol Z represents a dilution factor proposed to designate a dimensionless measure of sample dilution needed to reach some target effect (**10**).[5] For threshold work, the subscript "OL" represents the dilution at which the odor reaches a limit that corresponds to the best-estimate threshold.

X1.6 *Additional examples*—References (**11-20**) contain examples of thresholds determined according to this practice or by equivalent methods.

[5] The dilution factor, Z, is used in modest honor of H. Zwaardemaker, a Dutch scientist and early investigator in olfactometry. Alternate terminology in use: Dilution-to-Threshold Ratio (D/T or D-T); Odor Unit (OU); Effective Dose (ED).

REFERENCES

(1) Brown, D. G. W., et al., *Journal of the American Society of Brewing Chemicals*, Vol 36, No. 73, 1978.

(2) *Manual on Sensory Testing Methods, ASTM STP 434*, Am. Soc. Testing Mats., p. 11, section (e) for coding procedures.

(3) Baker, R. A., *Annals of New York Academy of Sciences*, Vol 116, p. 495, 1964.

(4) *Compilation of Odor and Taste Threshold Values Data, ASTM DS 48A*, Am. Soc. Testing Mats., 1978.

(5) Dravnieks, A., Schmidtsdorff, W., and Meilgaard, M., *Journal of the Air Pollution Control Association*, Vol 36, p. 900, 1986.

(6) Meilgaard, M. C., Reid, D. S., and Wyborski, K. A., *Journal of the American Society of Brewing Chemists*, Vol 40, p. 119, 1982.

(7) German Standard VDI 3881, Part 1. *Olfactometry. Odour Threshold Determination Fundamentals*. Verein Deutscher Ingenieure, VDI-Verlag GmbH, Düsseldorf, 1986, pp. 25–27.

(8) Morrison, G. R., *Journal of the Institute of Brewing*, Vol 88, pp. 167 and 170, 1982.

(9) Polta, R. C., and Jacobson, R. L., (Metropolitan Waste Control Commission, Minneapolis/St. Paul). Letter to A. Dravnieks, May 19, 1986, on file with Subcommittee E18.04.25.

(10) Turk, A., "Expressions of Gaseous Concentration and Dilution Ratios," *Atmospheric Environment*, Vol 7, p. 967, 1973.

(11) Amoore, J. E., Venstrom, D., and Davis, A. R., *Perceptual and Motor Skills*, Vol 26, p. 143, 1968. (thresholds in solutions)

(12) Guadagni, D. G., Buttery, R. G., and Okano, S., *Journal of the Science of Food and Agriculture*, Vol 14, p. 761, 1963. (thresholds in solutions)

(13) Hertz, J., Cain, W. S., Bartoshuk, L. M., and Dolan, T. F., *Physiology and Behavior*, Vol 14, p. 89, 1975. (thresholds in water solution)

(14) Dravnieks, A., *Annals of New York Academy of Sciences*, Vol 237, p. 144, 1974. (thresholds in air)

(15) Dravnieks, A., and Prokop, *Journal of the Air Pollution Control Association*, Vol 25, p. 28, 1975.

(16) Engen, T., *Perceptual and Motor Skills*, Vol 10, p. 195, 1960.

(17) Jones, F. N., *American Journal of Psychology*, Vol 69, p. 672, 1956. (general)

(18) Cederlöf, R., Edfors, M. L., Friberg, L., and Lindvall, T., *Journal of the Technical Association of the Pulp and Paper Industry*, Vol 48, p. 405, 1965. (thresholds in air)

(19) Meilgaard, M. C., *Technical Quarterly, Master Brewer's Association of the Americas*, Vol 12, p. 151, 1975. (thresholds in beer)

(20) Salo, P., Nykänen, L., and Suomalainen, H., *Journal of Food Science*, Vol 37, p. 394, 1972. (thresholds in alcohol-water mixture)